Edition: BP 2025 (Ph. Eur. 11.6 update)

Pyrethrum Extract

General Notices

DEFINITION

Pyrethrum Extract is prepared from Pyrethrum Flower.

Extemporaneous preparation

Exhaust Pyrethrum Flower, in *coarse powder*, by percolation with a suitable hydrocarbon solvent; remove the solvent and concentrate at a low temperature. The resulting product may be decolourised by a suitable procedure. Determine the proportion of pyrethrins in a portion of the extract by the Assay. To the remainder add, if necessary, sufficient Light Liquid Paraffin or *deodorised kerosene* to produce an extract of the required strength.

The extract complies with the requirements for Labelling stated under Extracts and with the following requirements.

Content of pyrethrins

24.5% to 25.5% w/w, of which not less than half consists of pyrethrin I.

CHARACTERISTICS

A dark olive green or brown viscous liquid or, if decolourised, a pale amber liquid.

ASSAY

To 0.5 g of the well-mixed extract add 20 mL of 0.5M *ethanolic potassium hydroxide* and boil under a reflux condenser for 45 minutes. Transfer the solution to a beaker and wash the flask with sufficient hot *water*, adding the washings to the beaker, to produce a total volume of 200 mL. Boil until the volume is reduced to 150 mL, cool rapidly and transfer the solution to a stoppered flask, washing the beaker with three 20 mL quantities of *water* and transferring any gummy residue to the flask. Add 1 g of diatomaceous earth (Filtercel is suitable) and 10 mL of *barium chloride solution*, swirl gently and add sufficient *water* to produce 250 mL. Stopper the flask, shake vigorously until the separating liquid is clear and filter the suspension through a filter paper (Whatman No. 1 is suitable).

If the pyrethrum extract is coloured, carry out the following preliminary treatment. Transfer 0.5 g of the well-mixed extract to a stoppered flask, add 50 mL of *aromatic-free petroleum spirit (boiling range, 40° to 60°)*, swirl, add 1 g of diatomaceous earth (Filtercel is suitable), swirl to mix completely, stopper the flask and allow to stand at 20° to 22° for 16 hours. Mix the contents of the flask thoroughly, filter with gentle suction through a sintered-glass filter (ISO 4793, porosity grade 4, is suitable) and wash the residue with five 10 mL quantities of *aromatic-free petroleum spirit (boiling range, 40° to 60°)*. Remove the solvent from the combined filtrate and washings and evaporate to a volume of 1 to 2 mL. Add 20 mL of 0.5M *ethanolic potassium hydroxide* and boil under a reflux condenser for 45 minutes. Transfer the solution to a beaker and wash the flask with sufficient hot *water*, adding the washings to the beaker, to produce a total volume of 200 mL. Boil until the volume is reduced to 150 mL, cool rapidly and transfer the solution to a stoppered flask, washing the beaker with three 20 mL quantities of *water* and transferring any gummy residue to the flask. Add 1 g of diatomaceous earth (Filtercel is suitable) and 10 mL of *barium chloride solution*, swirl gently and add sufficient *water* to produce 250 mL. Stopper the flask,

shake vigorously until the separating liquid is clear and filter the suspension through a filter paper (Whatman No. 1 is suitable).

For pyrethrin I

Transfer 200 mL of the filtrate to a separating funnel, rinsing the measuring vessel with two 5 mL quantities of *water*, and add 0.05 mL of *phenolphthalein solution R1*. Neutralise the solution by the drop wise addition of *hydrochloric acid* and add 1 mL of *hydrochloric acid* in excess. Add 5 mL of a saturated solution of *sodium chloride* and 50 mL of *aromatic-free petroleum spirit (boiling range, 40° to 60°)*, shake vigorously for 1 minute, allow to separate, remove and retain the lower layer. Filter the petroleum spirit extract through absorbent cotton into a second separating funnel containing 10 mL of *water*. Return the aqueous layer to the first separating funnel and repeat the extraction with 50 mL and then with 25 mL of *aromatic-free petroleum spirit (boiling range, 40° to 60°)*, reserving the aqueous layer for the assay of pyrethrin II, and filtering the petroleum spirit extracts through the same absorbent cotton into the second separating funnel. Shake the combined petroleum spirit extracts and water for about 30 seconds and allow to separate; remove the lower layer and add it to the aqueous liquid reserved for the assay of pyrethrin II. Wash the combined petroleum spirit extracts with a further 10 mL of *water*, adding the washings to the reserved aqueous liquid.

To the petroleum spirit extracts add 5 mL of 0.1M *sodium hydroxide*, shake vigorously for 1 minute, allow to separate and remove the clear lower layer, washing the stem of the separating funnel with 1 mL of *water*. Repeat the extraction by shaking for about 30 seconds with two quantities of 2.5 mL and 1.5 mL of 0.1M *sodium hydroxide* and add the extracts to the alkaline extract. Add to the flask 10 mL of *mercury(II) sulfate solution*, stopper, swirl and allow to stand in the dark at 25°± 0.5° for exactly 60 minutes after the addition of the mercury(II) sulfate solution. Add 20 mL of *acetone* and 3 mL of a saturated solution of *sodium chloride*, heat to boiling on a water bath, allow the precipitate to settle and decant the supernatant liquid through a filter paper (Whatman No. 1 is suitable), retaining most of the precipitate in the flask. Wash the precipitate with 10 mL of *acetone*, again boil, allow to settle and decant through the same filter paper. Repeat the washing and decanting with three 10 mL quantities of hot *chloroform*. Transfer the filter paper to the flask, add 50 mL of a cooled mixture of three volumes of *hydrochloric acid* and two volumes of *water*, 1 mL of *strong iodine monochloride reagent* and 6 mL of *chloroform*. Titrate with *0.01M potassium iodate VS*, running almost all the required volume of titrant into the flask in one portion. Continue the titration, shaking the flask vigorously for 30 seconds after each addition of the titrant, until the chloroform is colourless. Repeat the operation without the extract; the difference between the titrations represents the amount of potassium iodate required. Each mL of *0.01M potassium iodate VS* is equivalent to 5.7 mg of pyrethrin I.

For pyrethrin II

Transfer the combined aqueous liquids reserved in the Assay for pyrethrin I to a beaker, cover with a watch glass and evaporate to 50 mL within 35 to 45 minutes. Cool, washing the underside of the watch glass with not more than 5 mL of *water* and adding the washings to the beaker. Filter through absorbent cotton into a separating funnel, washing with successive quantities of 10, 7.5, 7.5, 5 and 5 mL of *water*. Saturate the aqueous liquid with *sodium chloride*, add 10 mL of *hydrochloric acid* and 50 mL of *ether*, shake for 1 minute, allow to separate, and remove the lower layer. Repeat the extraction successively with 50, 25 and 25 mL of *ether*. Wash the combined ether extracts with three 10 mL quantities of a saturated solution of *sodium chloride* and transfer the ether layer to a flask with the aid of 10 mL of *ether*. Remove the bulk of the ether by distillation and remove the remainder with a gentle current of air and dry the residue at 100° for 10 minutes, removing any residual acid fumes with a gentle current of air. Add 2 mL of *ethanol (96%)* previously neutralised to *phenolphthalein solution R1* and 0.05 mL of *phenolphthalein solution R1*, swirl to dissolve the residue, add 20 mL of *carbon dioxide-free water* and titrate rapidly with *0.02M sodium hydroxide VS* until the colour changes to brownish pink and persists for 30 seconds, keeping the flask stoppered between additions of alkali. Repeat the operation using the aqueous liquid reserved for the repeat operation in the Assay for pyrethrin I. The difference between the titrations represents the volume of *0.02M sodium hydroxide VS* required. Each mL of *0.02M sodium hydroxide VS* is equivalent to 3.74 mg of pyrethrin II.

STORAGE

Pyrethrum Extract should be kept in a well-filled container, protected from light and should be thoroughly stirred before use.